



## Materials Research Department annual report 2001

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# **Materials Research Department**

## **Annual Report 2001**

*Published by the Materials Research Department*

The background of the cover features a series of horizontal, wavy bands in shades of grey and olive green. A central, slightly tilted rectangular area contains a photograph of a green, textured surface, possibly a material sample, with a small, bright orange spot visible on the right side.

Risø National Laboratory

August 2002

Risø-R-1312 (EN)



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## ***abstract***

Selected activities of the Materials Research Department at Risø National Laboratory during 2001 are described. The scientific work is described in 10 chapters and a survey is given of the Department's educational activities along with a list of published work. Furthermore, the main figures outlining the funding and expenditures of the Department are given and a list of staff members is included.

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Front cover: Planar fuel cell, see page 16

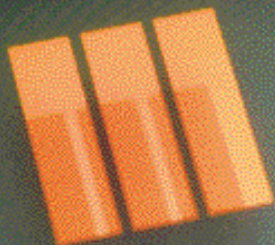
Contents cover: Materials testing, see page 8

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## 2001 - a very special year

The year 2001 was a very special, very exciting and a very successful year for the Materials Research Department. The Department entered the new year as a new department significantly larger than its two predecessors, the former Materials Research Department and the Condensed Matter Physics and Chemistry Department. The two departments, apart from the Polymer Chemistry, were merged into a new, strengthened department with about 130 persons in order to lead the materials research and solid state physics into a new era without access to the DR3 reactor, which very sadly had to be shut down permanently in September 2000. Furthermore, Robert Feidenhansl was appointed head of the new department from June 1st, succeeding Niels Hansen, who retired heading the old Materials Research Department for more than 30 years and Klaus Bechgaard, the former head of the Condensed Matter and Physics Department. In the evaluation of Risø that took place early in 2001, both 'old' departments were evaluated very positively and as "in many areas of world-leading class". It is going to be a challenging task for the new department to match this positive evaluation at the next evaluation in four years time.

The new Department includes basic science, applied science and technological development and has its focus on materials for future energy technologies. This includes materials for wind energy, especially non-destructive testing and composites, materials and research in superconductivity for energy transport, materials for Solid Oxide Fuel Cells (SOFC), materials for energy storage and lighter and stronger materials for energy savings. The department has one of its strongholds in the use of large scale facilities like synchrotron radiation, neutron scattering and electron microscopy for structural characterisation.

The department work has shown many highlights during 2001. Three of those have had exceptional importance and will be emphasized here.

Early in 2001 the department was selected by the Danish National Research Foundation to host the basic science centre 'Centre for Metal Structures in Four Dimensions' headed by Dr. Dorte Juul Jensen. The centre was awarded a grant amounting to 36 Mkr for a five years period. The aim of the centre is to study the relation between processing, properties and structure of metals. The heart of the centre will be the 3D X-Ray microscope that the Department has built at the synchrotron radiation facility ESRF in Grenoble and the new



*The Department at a turning point.*

transmission electron microscope which is currently being installed. With the aid of the 3D microscope using hard X-Ray energies the internal structure of a metal polycrystal can be described grain by grain as a function of processing. This has never been possible before and there is no doubt that the basic laws of deformation of metals have to be revised.

After the shut down of the DR3 reactor, the neutron scattering instruments at Risø needed a new home. Most of them were world-class instruments producing data for high quality publications. Fortunately, at the end of 2000, Risø made an agreement with the Paul-Scherrer Institute (PSI) in Switzerland that gave such a new home for three of the best instruments. Two were the inelastic scattering machines called RITA II and RITA I while the third was the highly productive small angle scattering instrument SANS. RITA II never made it into operation at DR3, and had to be modified under great time pressure by the technical staff of the department to fit the SINC spallation source at PSI. The transfer was completed successfully to the extent that the first data were presented already at the first Swiss-Danish neutron scattering user meeting held at PSI in November 2001. The SANS instrument will be transferred in April 2002 while the RITA I transfer will have to wait until 2003. Each of the instruments installed at the PSI returns 60 beam days to the Danish neutron scattering user community.

A significant milestone was reached after the summer by the agreement with the company Haldor Topsøe A/S concerning future development of Solid Oxide Fuel Cells (SOFC) materials and technology for commercialisation. The department has for more than ten years performed research and development in materials for SOFC sponsored by national energy programs (energistyrelsen, PSO), the EU and Haldor Topsøe A/S. During that period the department has become one of the internationally leading institutions in the field. With a new design of the cell, the potential cost of a stack could be significantly reduced and hence an important step towards commercial use was taken. However, the international competition is very strong and a range of new improvement on the materials and technology needs to be performed. This can only be achieved if the De-

partment in parallel develops its competence in basic understanding of the properties of ceramics and how to process them. The agreement with Haldor Topsøe A/S gives the long term perspective that will allow these developments to be undertaken for carrying a new Danish energy technology product on the market in five to ten years.

There are many other highlights' that could be mentioned. A few of them are described in more detail on the following pages, but by far not all. This year's annual report is different from previous annual reports from the two 'old' departments. We have decided to focus on only a few of the exciting scientific highlights of year 2001, but there are many more than presented here; just take a glance of the list of publications.

### Science and publications

Scientific impact is in the academic world often measured by the number of publications in international journals. The department has performed excellent here with 147 publications in international, refereed journals. Out of these 14 were in high ranked journals like Science or Physical Review Letters.

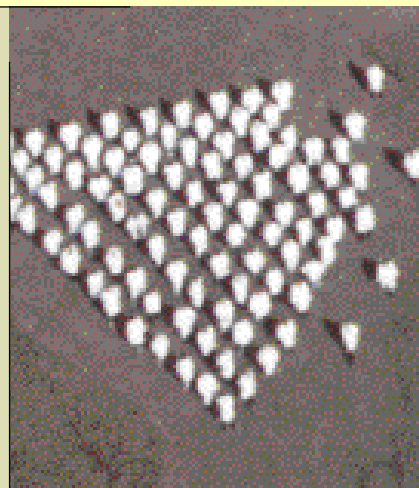
Dorte Juul Jensen was elected to be editor of the journal 'Scripta Materialia', one of the leading journals in materials science. This is a very prestigious responsibility for Dorte and we are honoured in the department that one of our staff members were elected.

Further, P.A.Lindgård has been elected to serve on the executive editorial board for Journal of Physics Condensed Matter.

The use of X-Ray synchrotron radiation has become a more mature field which has many applications in physics, chemistry and materials science. It has also set new standards for the understanding of x-ray scattering, so the time a new text book on fundamental X-Ray physics was ripe. Des McMorro, research specialist in the department, has together with prof. Jens Als-Nielsen from the University of Copenhagen written a new text book 'Elements of Modern X-Ray Physics', which was published early in 2001 and already has sold more than thousand copies.

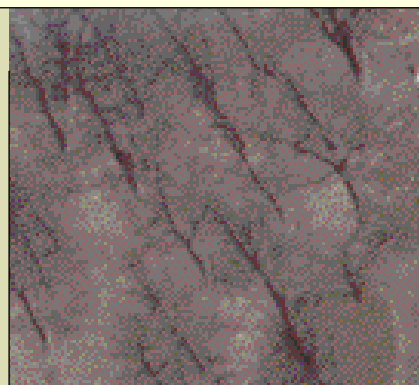
## Education

Basic research should not be undertaken without a close link to education. The department is engaged on all levels. 6 Ph.D. students got their degree in 2001 and more will follow in 2002. We had 18 Ph.D. students working in all research areas covered by the department. Furthermore we had 5 master students, out of which one got degree in 2001. 7 Summer students spend their summer working in the department and we hope to see them all later as master or Ph.D. student. Finally, the staff has been engaged in a number of lectures and classes at the universities in the Copenhagen area.



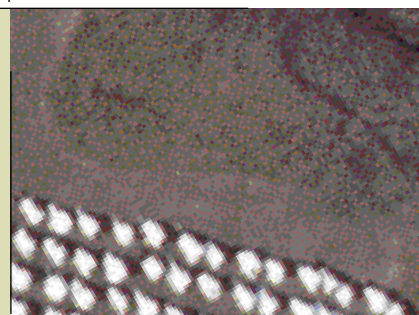
## Innovation

Bringing the research to applications is important for the benefit of society, the society that funds our research. Apart from the agreement with Haldor Topsøe A/S, the department has had collaboration with many others industries, especially in the area of wind energy and superconductivity. The department has many patents and is planning to put more emphasis on patent policy in the coming years.



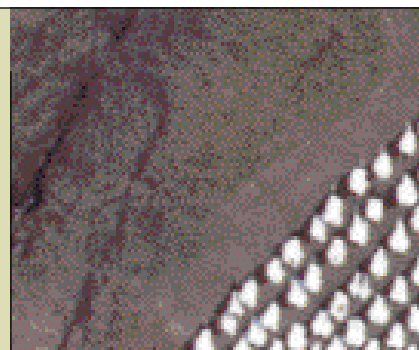
## Staff

The department engages about 130 people, out of which 45 are senior staff members, 10 scientists, 45 on the technical and office staff, 30 Ph.D. students and Post Docs. The age distribution is very flat, which is important for the exchange of knowledge and expertise and for the social and academic atmosphere.



## Economy

The department had a total turnover in 2001 of 121 Mkr. About half the income is external with 43 Mkr from program research and 15 Mkr from market-driven commercial income and 63 Mkr from Risø. The expenditure is to a large extent on salaries with 46 Mkr, overhead back to the Risø infrastructure 42 Mkr, investments 14 Mkr.







*The best steels available for cold forging applications are the highly alloyed powder metallurgical (P/M) steel grades. Ph.D.-student Christian Højerslev is visually investigating the fracture surface of cold forging tool (a punch) made of such a steel, with the aim to locate the crack initiating site.*

## A multiscale view of the mechanical behaviour of materials

As all other materials properties the mechanical properties are ultimately determined by the interaction of the constituent atoms. However, this does not mean that we have to think in terms of atoms whenever we try to understand the mechanical properties. On the contrary there is a Chinese-box system of levels at which we may view the mechanical behaviour of materials. Some processes are best understood at the atomic level (at the scale of fractions of nanometers). Other processes are best understood at the level of individual dislocations (at the scale of many nanometers) or groups of dislocations (typically at the scale of fractions of micrometers). Other processes yet are best understood at the grain level (typically at the scale of micrometers or many micrometers). However, sometimes we may “jump” the scales. In nanostructured materials the grain size may come down to few nanometers. In the first case story on the next page we deal with dislocation walls with nanometer-scale wall distances. Superimposed on the levels defined by the lattice defects are the levels defined by precipitates and phase distribution which may range from the nanometer scale to the micrometer scale.

At the Materials Research Department we deal with mechanical properties viewed at all the different scales. At the atomic scale, for instance, we have in collaboration with Department of Physics at the Technical University of Denmark determined the activation energy for cross slip of a jogged screw dislocation. In collaboration with Department of Manufacturing Engineering and Management at the Technical University we have investigated the initiation and growth of cracks (starting at the nanometer scale and ending at the mm scale) in cyclically loaded high-strength tool steels with nanoscale precipitates. This is described in the second case story on page 5.

*Torben Leffers*

### Graded nanostructures produced by sliding

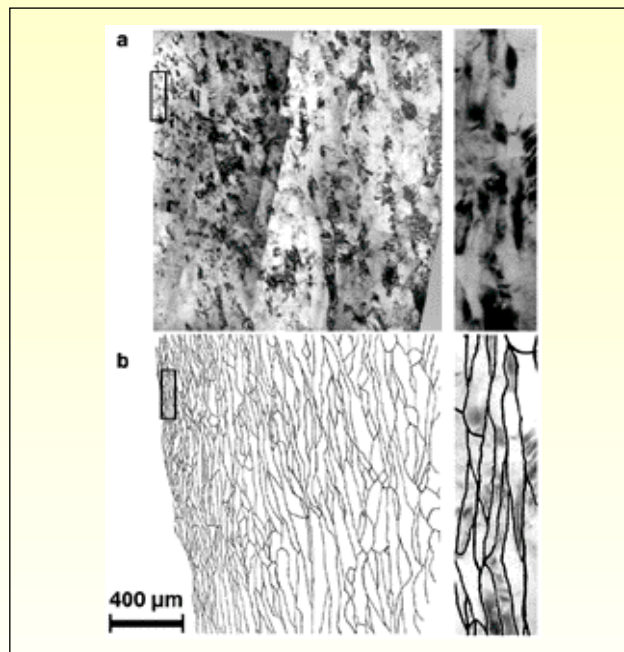
In collaboration with Sandia National Laboratories in USA "friction experiments" were carried out on copper specimens. The specimens were exposed to a hardened steel plate, which was pressed against the specimens and slid along the surface. This produced very high strains in the copper surface layer. Transmission electron microscopy of the surface layers of the specimens revealed a microstructure consisting of dislocation walls parallel to the surface with a wall spacing of  $\sim 10\text{nm}$  at the very surface and increasing wall spacing with increasing depth below the surface. It is known from studies of bulk deformation that high cumulative strains (up to von Mises equivalent strains of  $\sim 5\text{--}10$ ) can lead to fine microstructures down to a scale of  $\sim 100\text{nm}$ . Extrapolation of the experimental relation between the scale of the microstructure and the cumulative strain for bulk processes indicates that the strain at the very surface of the copper specimens in the friction experiments is  $>100$ .

If the normalized distributions of the wall spacings (wall spacing divided by average wall spacing) for different depths below the surface in the samples from the friction experiments and the normalized distributions for bulk nickel rolled to 70% and 98% reduction are plotted together they make up one single distribution for average wall spacings from  $\sim 10\text{nm}$  to  $\sim 300\text{nm}$  and local equivalent von Mises strains from  $\sim 1$  to  $\sim 100$ . When all the points are part of one single distribution, this is a strong indication that wall formation for this wide range of conditions is governed by one single process – a truly multiscale process.

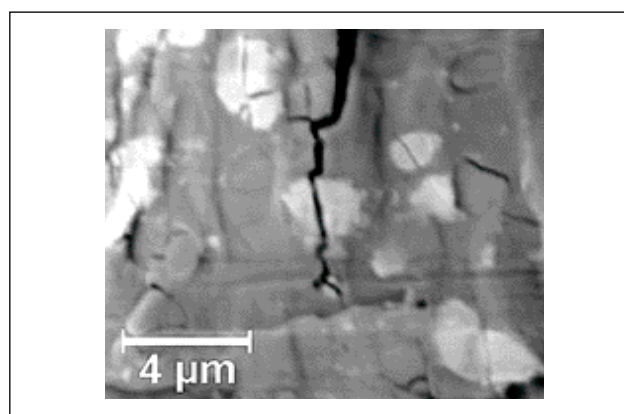
### Fatigue cracks in tool steels

A high speed tool steel was subjected to four point bending fatigue tests with the aim of studying the influence of microstructure on crack initiation and crack growth. The steel is a high strength composite material consisting of a martensitic matrix with more than 20 vol.% carbides and with a yield strength exceeding 2 GPa. The steel has tensile stresses in the matrix and compressive stresses in the carbides due to dissimilar thermal expansion coefficients of and Young's moduli in the constituent phases. As a consequence the larger carbides ( $0.5\text{--}2\text{ }\mu\text{m}$ ) fracture when subjected to loads higher than approximately 80% of the yield strength. Transmission electron microscopy of the matrix reveals no microstructural changes during deformation. This suggests, that the matrix contains smaller obstacles, which prevent dislocation movements and thus disables stress relaxation of the carbides. It is likely that the obstacles are nanosized carbides, but this needs to be confirmed with the new 300 kV FEG-TEM. On notched specimens a carbide damage zone exists, and the size of this zone increases

with increasing load amplitude whereas it decreases with increasing sharpness of the notch. The fractured carbides within the zone appear to be associated with crack nucleation and they may also have an effect on crack growth.



Graded microstructures produced by friction deformation. (a) TEM micrograph and (b) tracing of extended boundaries. The left side is coincident with the surface. Rectangular boxes show the surface layer with 12 nm average boundary spacing.



SEM micrograph showing a fatigue crack in a tool steel. Carbides appear as light phases on the micrograph, and several changes in the crack path are observed in the vicinity of fractured carbides. A crack approaching a fractured carbide are expected to be attracted, whereas intact carbides are expected to repel the crack, because the Young's moduli of the carbides are significantly larger than that of the matrix. This suggests, that the carbides fractured before the crack approach, and therefore they promote, rather than inhibit, the crack growth.

## Metal Structures

As metals have been used and optimized for more than 3000 years, one could believe that everything is known about metals. However, this is indeed not the case.

Advances in metal science have generally relied on developing relationships between structure and properties. It is, however, far from trivial to characterize the structure in sufficient detail to understand the fundamentals of its evolution. "Quantum leaps" in this understanding have generally followed the development of new characterization techniques, e.g. electron microscopy.

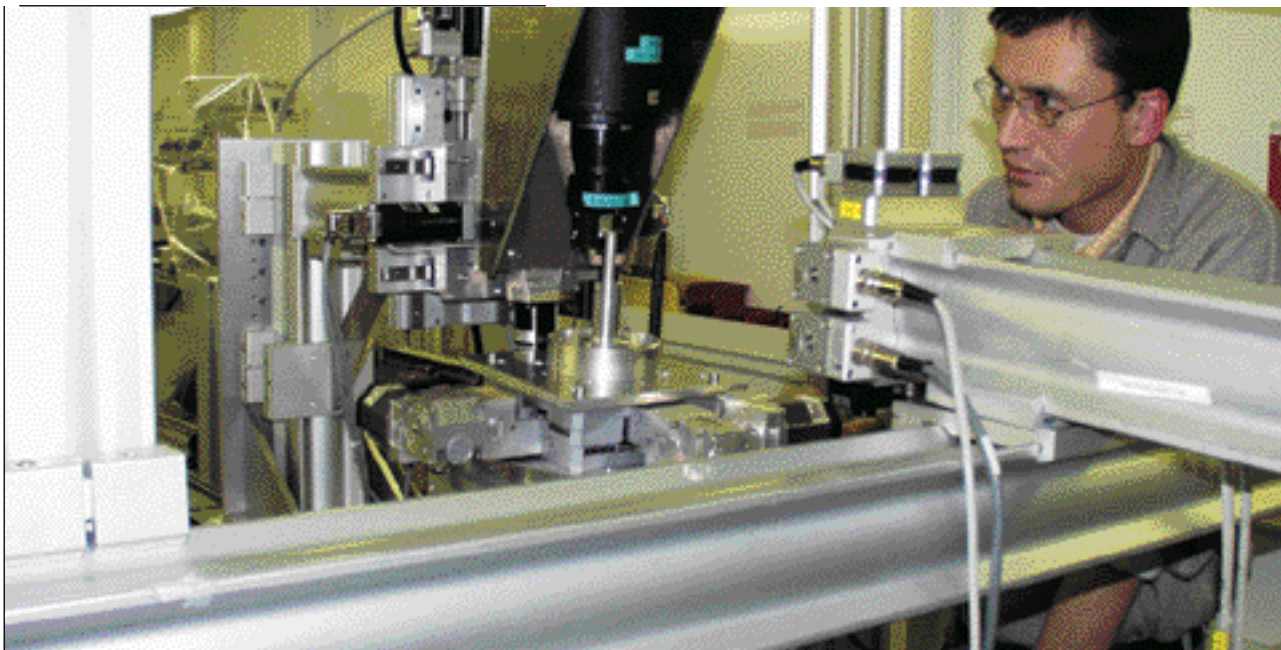
Traditional techniques for structural characterization are either limited to detailed inspection in 2D or coarse-scale (mm-range) inspections in 3D. The 2D techniques can be used only for static characterizations at discrete processing intervals (i.e. post mortem analysis) and not for in-situ characterization of the dynamics of structural development. The coarse-scale techniques, on the other hand, cannot reveal the individual "building blocks" of the structure, e.g. defects, dislocation boundaries or nuclei, and, thus, only average characteristics may be derived.

X-Ray diffraction microscopy offers the possibility of time resolved three-dimensional mapping of structures to micrometer scale.

During 2001, the Centre for Fundamental Research: Metal Structures in Four Dimensions, was started supported by the Danish National Research Foundation by 36.572.000 DKR. The overall idea is to explore the heterogeneous structure of metallic materials and understand how this responds over time to changes in stimuli such as stress and temperature. A key technique will be the 3 Dimensional X-Ray Diffraction (3DXRD) microscopy but also electron microscopy will be an extremely important technique. The work will include experimental characterizations as well as theoretical modelling on several length scales. There will be five lines of research focusing on i) improvements of the 3DXRD microscope, ii) dislocations, iii) boundary migrations, iv) nano-scale structures and v) multiphase alloys.

The overall goal of the new Centre is primarily to significantly advance the knowledge and understanding of metal structures, but also the development of new experimental techniques and visualisation tools to be used by a broader community than metallurgists are major goals.

*Dorte Juul Jensen*



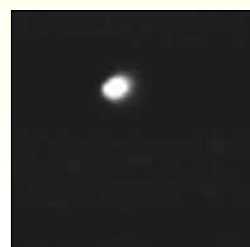
*Søren Fæster Nielsen is carefully lining up the 3 dimensional x-ray microscope for grain mapping.*

### Rotation of individual grains

The new 3 dimensional X-ray technique is the only existing technique with sufficient penetration depth and spatial resolution to follow structural changes in metals during deformation on the scale of individual grains. During deformation, the individual grains change their shape and must rotate, i.e. change the orientation of their crystallographic lattice, to stay in contact with the neighboring grains.

For the first time, this rotation has been monitored for about 100 grains deeply embedded in an aluminum sample subjected to tensile deformation. Initially, the grains were randomly distributed in orientation space. During deformation, the grains rotate towards the stable tensile texture components. There is a clear correlation between rotation direction and the initial orientation of the grains, indicating that interaction with their different neighbors does not dominate the rotation behavior.

From the point of fundamental research, this is very important new experimental information. Until now the experimental basis for this understanding has been limited to studies of similar but not identical samples before and after deformation which does not give any information on the rotation of individual grains. This information is essential to identify the factors controlling the rotations. The research also has technological importance because many properties, e.g. mechanical and magnetic, depend on the ensemble of crystallographic orientations present in a material.



*Raw images from the 3DXRD microscope showing diffraction spots from aluminium. The inserts show a spot from the same grain before and after 6% elongation. It is seen that the spot spreads out and moves as the lattice rotates.*



## Material Mechanics

Material Mechanics deals with characterisation, measuring, and modelling of mechanical properties in a variety of materials and composites under static and dynamic loading conditions. The materials include both new materials and industrial materials and the aim is to create basic knowledge of the relation between micro mechanical behaviour and the material properties to be used in design and in lifetime and health analysis of components and structures. The mechanical pro-

perties and the basic knowledge of the material microstructural behaviour are also considered in tailoring new materials for optimised use both in new and conventional applications.

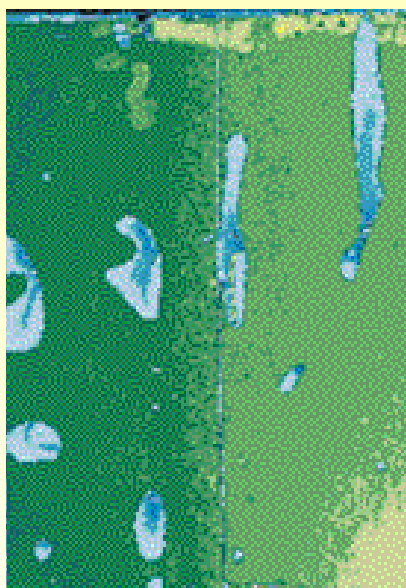
The main research topics are analysis of material degradation and development of constitutive equations to simulate this behaviour. Projects are dealing with investigation of failure mechanisms in composites and sandwich materials by studying fracture mechanics and the dynamics of crack growth. Interface mechanics are in special focus as damage evolution and fracture of fibre composite materials are originated in the micro structural interfaces. Through these research topics failure modes are studied based on fracture mechanics, damage mechanics and bridging effects.

Modelling mechanical behaviour requires analytical models based on fundamental topics in solid mechanics and development of constitutive equations for the materials. FEM models are set-up using the ANSYS code. The mechanical testing activities cover all types of standard testing (static, fatigue, creep and impact). In addition to carrying out specialised testing on any type of material or composite the laboratory is also an accredited test laboratory (by Danish Accreditation, DANAK). Furthermore, specialised demonstration and verification tests are carried out on components. The non-destructive characterisation activities cover X-ray imaging, ultrasonic scanning, acoustic emission and thermography. Activities are focused on integration of surveillance techniques in general health monitoring systems.

The Department participates in a variety of national and international research and development projects, within materials mechanics. The active projects during 2001 have focused on: Manufacturing and material development; Characterisation and Design; Surveillance and Damage Detection. In addition to these activities there are commercial projects, which are confidential research and development, and are directly supported by Danish and international companies.



*Laser extensometer used in testing ultra high strength tool materials. The transverse extensometer is used for determining lateral strain and Poissons ratio.*



*A specimen consisting of two 2 mm aluminium plates (70 x 100 mm) was bonded by a 0.2 mm adhesion layer. The ultrasonic scanning (left picture) of the interface aluminium/adhesion showed areas with bad adhesion or no adhesion. The photograph (right picture) shows the adhesion surface of one of the plates after the plates were separated - there are no adhesives in the light areas. There is a very good correspondence between the two pictures demonstrating the accuracy of the ultrasonic inspection.*

## COLT

Cold forging is the most efficient technology for mass production of high precision parts for transport, mechanical and electrical industries. Cold forging tools operate under extreme conditions where slight variations in process load and material strength have a strong impact on tool failure. More than 80% of tools in cold forging fail because of fatigue damage due to cyclic plasticity. The Department participates in an EU/BRITE project dealing with "Improvement of Service Life and Reliability of Cold Forging Tools with respect to Fatigue Damage due to Cyclic Plasticity" (acronym COLT). The main objective of this project is to develop a methodology to increase tool life and to reduce its scatter. A large number of fatigue tests have been carried out on ultra high strength tool steels supplied by Böhler Edelstahl in Austria. This has provided important information about the mechanical behaviour of these steels, and in combination with subsequent microstructural characterization, it has been possible to identify the fundamental deterioration mechanisms. These mechanisms form the basis of microstructural modeling of the tool steels. Other partners involved in this project are Thyssen-Krupp-Presta, Strecon A/S, Rockfield Software Ltd, Centre for Computational Continuum Mechanics (C3M) and Univeristy of Erlangen-Nuernberg.

## CLEA

In the Danish Center Project CLEA (Light and Elegant Constructions from Aluminium Using Adhesive Joining) the aim is to improve and increase the use of aluminium by the use of adhesion. Adhesion technology enables large degrees of freedom for creative industrial design and the use of aluminium results in up to 25-50% reduction of the total weight of a product. However, a prevalent scepticism exists towards the use of adhesive joining, in particular when it comes to the long-term strength of joints made by adhesion. The project aims to overcome the essential difficulties of technical, attitudinal and scientific nature, which prevent the industrial use of adhesive joining in loaded constructions and products. The Department contributes with development of non-destructive techniques for characterisation and control of adhesive joints. Also, a concept for calculation and prediction of the lifetime of the joints is developed. Other partners involved in the CLEA project are LG Trafik A/S, Bent Falk Design, 3M A/S, Hydro Aluminium Tønder, Technological Institute, The Architect and Engineer Schools in Århus and Aalborg University Esbjerg. More information (in Danish) can be found on [www.clea.dk](http://www.clea.dk).

*Povl Brøndsted*

## Composite Materials

To meet the energy demand several alternative methods for energy production have been exploited and developed during the last 20 years. Among the most prominent of these techniques is the wind energy, which has from the earliest time, i.e. since the mid-1970's, attracted particular interest in Denmark. One reason for this activity is the large resources of wind energy available in Denmark, both on-shore and offshore, caused by the relatively strong and constant wind in North-western Europe. Another reason for the technical development of windturbines in Denmark is the enthusiastic entrepreneurs, who, in particular in the early days of wind energy, started "from scratch" and produced small windturbines, which functioned with a minimum of performance problems. These early small windturbines were relatively sturdy and heavy, in particular the rotorblades were built from glass fibre composites with existing fabrication technology from the boat building industry. This resulted in rather heavy, but also very durable rotorblades, which functioned under the long time required for windturbines. Other countries initiated their wind energy activities on the basis of knowledge from the aircraft industry, and this turned out to be a much slower process for technical and market development. This led to Denmark taking a leading position in the practical and commercial wind energy field, a position, which has been maintained over the years. The several small entrepreneurs have been replaced by rather few large companies with activities both in Denmark and in foreign countries.

The typical windturbine, of Danish design, consists of a tower, with a cabin/nacelle on top, which supports the rotor and contains the gear, generator and braking system. The tower is made of typically steel or concrete, the cabin is composed of steel, aluminium or composite material, and the rotorblades are exclusively made of composite materials and in some cases of wood or wood-related materials.

The materials problems and the related research are focused on the rotorblades. The most common materials used since the initiation of modern wind energy technology are composite materials based on glass fibres and polyester-matrix. These composites contain about 50 volume per cent fibres; these are arranged parallel to the major loading direction for the primary load bearing structural components. In other parts of the rotorblade with secondary structures, multi-oriented fibres, typically 0°, 45°, 90° - configurations, are used. The outer aerodynamic shells of the rotorblades are sandwich-constructions with core materials of balsa-wood.

These composite materials have good mechanical properties, in particular stiffness and fatigue strength, for present day rotorblades, which have lengths up to about 35 m with a (net-) weight of ca. 6000 kg. The manufacturing of such rotorblades requires manual labour and is partly automated.



Structural health monitoring during full scale wind mill blade test at the Risø Sparkær test facility. staff from the Department is Bent Sørensen (3rd from left), Malcom McGugan (4th from left) and Christian Debel on the right.



*Acoustic emission monitoring of a crack in a wind mill blade. The detector is the device mounted on the right.*

Development in materials and processing are taking place. The introduction of carbon fibres, epoxy-matrix and prepreg technology is in progress at the manufacturers. The use of thermoplastic polymer matrices is being investigated, and combinations of glass/polyester composites in combination with wood (veneers) and carbon fibres are under development.

The recent materials developments are necessary in order to reduce the (relative) weight of the rotorblades by using materials of lower density. This is dictated by the increased dominance of the gravity-forces experienced by the future very large rotorblades and windturbines of megawatt sizes.

Lower composite density can be achieved by using fibres of lower density than that of glass; candidates are carbon fibres and cellulosic fibres, of which carbon fibres are industrially available with a price still somewhat too high, and cellulosic fibres are under development from plants, typically flax and hemp. The potential advantage for a large rotorblade will be a (net-) weight, which is between 50% and 70% of the weight of a glass/polyester-based rotorblade.

The large and long rotorblades up to 60 m, or even longer, would be very flexible if they were based on glass/polyester composites; this might cause practical problems in relation to the blades passing the tower during rotation, and design problems in relation to aero-elasticity. An improved stiffness of the

rotorblades can be achieved with the use of high stiffness carbon fibres in the composite. At present, this line of research and development is pursued in the form of hybrid composites, containing a combination of glass-fibres and carbon fibres. This, in addition, gives a weight advantage.

The damage developed in composite materials during loading, in particular fatigue loading, is of importance for the state of "health" of the material, and thus for its life time before un-acceptable damage or final failure. Studies have been intensified in recent years on the development of damage and its relation to composite materials structure and fatigue loading histories. In parallel, techniques in particular non-destructive techniques, are under development to monitor damage during the functioning period of rotorblades. Research is in progress to understand the link between damage and mechanical properties/life time of composite, and to establish the link between monitoring signals and state of damage. This structural health monitoring aims at establishing a procedure to continuously record the "state of health" of the rotor, so that actions (accept, repair, replacement) can be initiated in good time before irreversible damage develops.

*Hans Lilholt*



## Nano-structured materials

The properties of nano-structured materials is a challenging field in which the typical distances are of the nano-meter scale: Larger than the atoms, but much smaller than the crystal grains. In structures of the nanometer size one may encounter completely new, exiting, and useful material properties. Basic research on this topic is mostly labelled nano-science, while the word nano-technology stands for applied research. A common feature of all parts of nano-science and -technology is that the nano-sized materials are extremely difficult to create artificially, and it is often necessary to rely on Nature's own ways of self-assembly of atoms and molecules to create the nano-structures.

In the Department we deal with the inorganic part of nano-science and nano-technology, such as nano-structures in bulk materials. One example is the bulk amorphous alloys, which unique softening properties may be used to forge nano-scale objects. Another method of obtaining nano-sized patterns is by silicon bonding. By placing a few-nano-meter thick "peel" of Si crystal onto bulk Si, one may obtain a regular modulation pattern, which modulation distance depends on the misorientation angle between the crystalline directions of the "peel" and the substrate. Also the magnetic properties of bulk amorphous alloys are of interest. In NdFeCoAl alloys, we have found magnetic properties ranging from very hard (for permanent magnets) to very soft (for transformers).



*In the supercooled liquid state the amorphous alloy becomes remarkably soft. Nini Pryds is pressing an  $Mg_{60}Cu_{30}Y_{10}$  alloy lightly with a tool at approx. 160 °C.*



*The subsequent impression is shown after cooling to room temperature. Only low forces are needed to shape the material and the amorphous properties are maintained.*

Nano-sized particles have a very high surface-to-volume ratio, which makes them well suited for catalysis purposes. We study these properties in collaboration with Haldor Topsøe A/S. In the Department we further study magnetic and superconducting properties of nano-sized particles and how the properties vary with particle size, shape, and composition. We will extend this activity to study the properties of nano-composite materials, formed by a mixture of different nano-particles, or by inserting magnetic nano-particles in a non-magnetic matrix. This work is performed in collaboration with the Technical University of Denmark and University of Copenhagen.

An obvious way of studying nano-structured materials is by means of scanning probe microscopy. Here, one can map out the structure and the electric and magnetic properties of a small surface area or of individual particles. Complementary information may be found by X-ray and neutron diffraction or electron microscopes, which gives indirect information of the structure of larger samples of nano-crystalline materials. Neutron scattering may further give information on the magnetic structure of materials – and on vibrations in the magnetic structure. All these methods are used in the research of the Department.

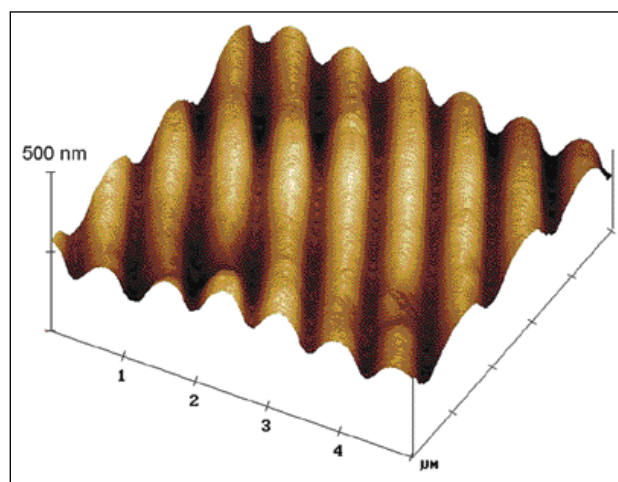
Kim Lefmann

### Amorphous alloys

Bulk amorphous alloys are metallic alloys in which crystallisation kinetics is sufficiently slow so that the amorphous atomic arrangement of the liquid is retained during ordinary metal casting operation. When heated, bulk amorphous alloys transform to a supercooled liquid state prior to crystallising and remarkable softening is associated with the transition from a hard amorphous glass to a viscous supercooled liquid (SCL). A bulk amorphous alloy with composition  $\text{Mg}_{60}\text{Cu}_{30}\text{Y}_{10}$  is being studied in the Materials Research Department at Risø and the softening behaviour of this material was evaluated during 2001. Mechanical property measurements revealed for the first time that the material transformed from a hard metallic glass to a soft viscous state (not unlike window putty) upon increasing the temperature from 140 °C to 170 °C. Such abrupt softening is a feature of bulk amorphous alloys, which has no parallel among conventional crystalline alloys. Since the amorphous atomic arrangement of the SCL state has no grain structure to induce heterogeneity of deformation, forging of bulk amorphous alloys in the SCL state was hypothesised to permit faithful replication of extremely detailed surface features. To test this hypothesis, samples of the  $\text{Mg}_{60}\text{Cu}_{30}\text{Y}_{10}$  were forged using dies having microscopic surface features, and the result of such microforging tests were evaluated by atomic force microscopy. The figure shows the exceptional fidelity of replication of a linear sine-wave grid

with a wavelength of 800 nm. Various applications of this microforging capability are being further explored, including extension of the microforging capability to the nano-scale.

John A. Wert



Atomic force microscopy image of amorphous  $\text{Mg}_{60}\text{Cu}_{30}\text{Y}_{10}$ . The surface structure was obtained by pressing a sine wave-shaped metal surface (produced by photo lithography) on to the supercooled liquid alloy. The photo shows the exceptional replication fidelity of the pressing operation.



## The Fuel Cell Programme

Since the introduction of fuel cell development in 1987, the Department's Fuel Cell Programme has gradually developed an international competence within fundamental research and technology, which is currently at a pre-commercial level. Presently, the programme is experiencing extensive growth, made possible through creating links between the research facility and commerce, in close co-operation with both national and international companies, and supported by Danish Energy Research Programmes and the EU. As part of this development, the programme has developed a project-orientated structure, achieving synergy between the competencies within specific work groups and the work required within national and international projects. This structure enables the individual work groups to specialise at different stages in a projects life cycle, for example within cell component development, cell testing, or stacking technology.

Risø has established a collaboration agreement with the international company Haldor Topsøe A/S to develop Solid Oxide Fuel Cell (SOFC) technology for commercial use. This agreement secures for the Department a minimum of 35 man-years of research and development into SOFC technologies over the next 5 years. The collaboration has a unique nature, with the ambition of having Risø and Topsøe as world leaders in solid oxide fuel cell technology and science. Haldor Topsøe A/S will be responsible for commercialisation, industrial production and marketing. Risø will primarily work with developing the

technology and participate in medium and long-term research to secure a leading international position for Danish fuel cell technologies.

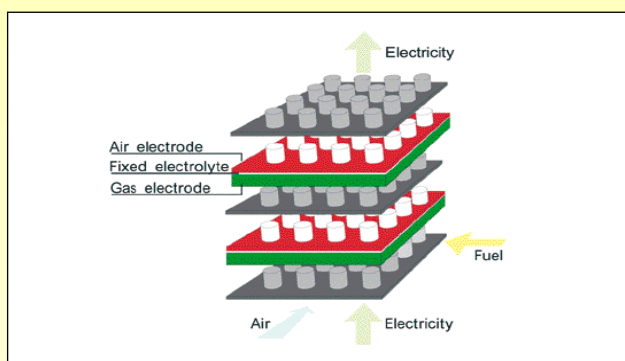
Solid Oxide Fuel Cells produce both electricity and heat, therefore, the technology offers attractive possibilities for heat production for individual houses, or for district heat production plants. Within the next 10 or 15 year period, fuel cell systems are expected to be used in industrial plants and power stations.

Moreover, there is increasing interest for their use as auxiliary power units (APU's) for vehicles and uninterrupted power supplies (UPS's).

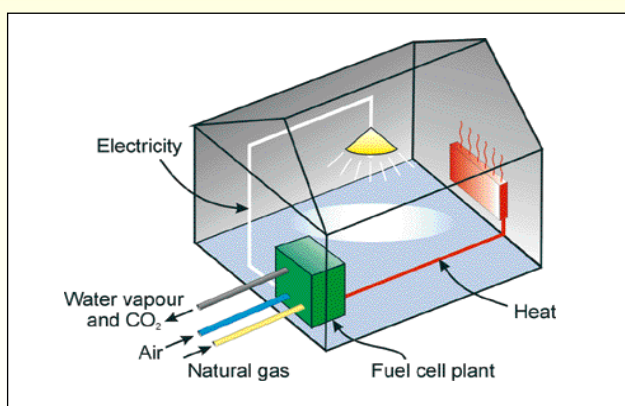
An important part of the commercialisation of fuel cell technology, through its collaboration with Haldor Topsøe A/S, the Department has established an experimental production plant to manufacture flat plate cells at Risø. The purpose of the plant is to scale-up existing laboratory production methods, build up the necessary know-how for establishing industrial production, and to develop non-destructive methods for production control. The plant will aim to produce homogenous cells for analysing and optimising cell life. Furthermore, a high production level will enable a number of cells to be used to develop and optimise Danish cell stacks and system technology, and to demonstrate Danish fuels cells to an external audience.



*An experimental production plant for the manufacture of anode-supported cells has been established at Risø in collaboration with Haldor Topsøe A/S. The plant comprises a 20 meter long continuous tape-caster that is used for the production of anode supports.*



A single cell delivers an output of close to 1 Volt, but a higher voltage can be achieved by stacking more unit cells on top of each other. These are interconnected by metallic components (black) and are separated by electron conductive cylinders (approximately 1 mm high) to enable gas flow.



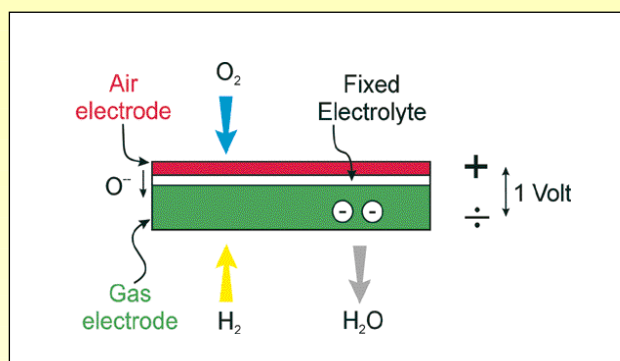
### How do fuel cells work?

Fuel cells work as an energy conversion device that generate electricity and heat by electrochemically combining a gaseous fuel and an oxidant (air), via an ion-conducting electrolyte. The dominating waste product is water. The benefits of fuel cells are the efficient, silent and environmentally friendly power generation possibilities at low price.

### Cell stacking

A single fuel cell delivers an output of a slightly under 1 volt. If the generated power is to be used effectively, many cells must be arranged in series, in a so-called 'cell stack'. The development of low cost and effective stacking techniques involves many challenges and problems in the area of materials science. Experimental facilities are in preparation for investigating these issues and testing new solutions.

The stack test houses are built to enable the testing of different cell sizes and stack configurations, with careful control of the experimental conditions. Novel equipment has been de-



The principle behind Solid Oxide Fuel Cells (SOFC): On the positive electrode (Red) the oxygen in the air reacts with electrons and creates negatively charged oxide ions. These migrate through the electrolyte (White) over to the negative gas electrode (Green). Here the oxide ions react with hydrogen and produce water. Individual cells produce a voltage of about one-volt in open circuit, but this decreases under electrical load, due to the cells internal electrical resistance. To reduce this loss, the internal resistance of the cell must be minimised by optimising electrodes and making the electrolyte as thin as possible. To reach high voltages prior to AC conversion, fuel cells can be arranged in series to create 'cell stacks'. The strengths of the fuel cell programme at Risø lie in the fundamental science and technology needed to create the individual cells and stacks.

In the future, fuel cells may provide both electricity and heat by a plant situated in each individual house.

veloped to observe and analyse stack operation under test conditions.

### Cell testing

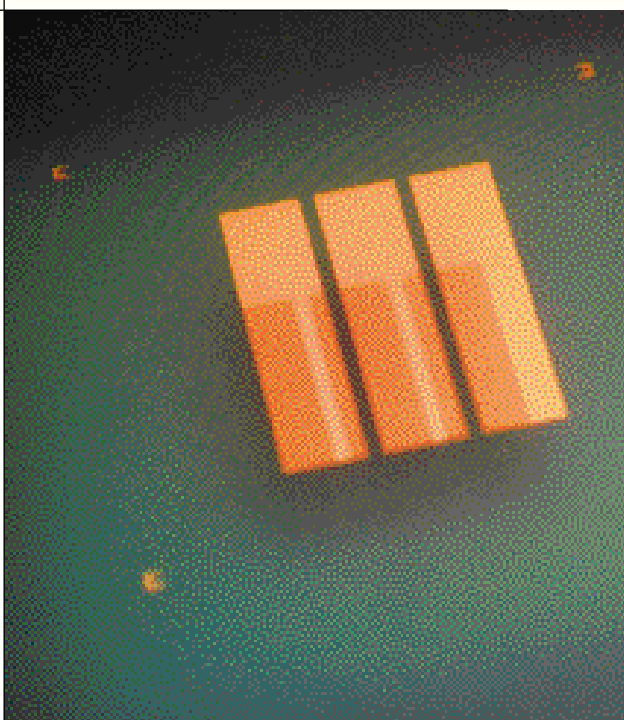
The in-house development and introduction of four 'cell test houses' has considerably advanced Risø's possibilities for cell testing, enabling efficient and accurate procedures for the testing of anode supported SOFCs. The new 'test house' provides better control over test parameters; the two gas compartments are sealed by glass seals and the rigs are equipped with  $pO_2$  (oxygen partial pressure) sensors at both the fuel inlet and outlet. This enables accurate determination of fuel utilisation and efficiency. Furthermore, the accurate operating temperature of the cell is determined by thermocouples in direct contact with the cell. These features allow improved accuracy in the description of cell operation.

[ to be continued on page 16 ]

[ continued from page 15 ]

### Integrated Planar Solid Oxide Fuel Cells

In parallel to the work on stacked cells described above, a significant effort is being made on an alternative cell concept. An EU-funded project is directed towards the development of an affordable Solid Oxide Fuel Cell of modular design, that can operate as a sub-MW standalone heat and power plant, or as a multi-MW system combining a SOFC and a gas turbine cycle. The technology is based on the Integrated Planar Solid Oxide Fuel Cell technology developed at Rolls Royce, UK and, due to the multi-functional capacity of the cells, the project is termed MF-SOFC. The project is co-ordinated by Rolls-Royce, and also involves Imperial College UK, Advanced Ceramics Limited UK, and Gaz de France. The scale-up in rating and production volume calls for high reliability and durability, while maintaining low production costs. Fabrication costs are kept low by using well established thick film fabrication techniques, including silk-screen printing of the majority of cell components. Risø's tasks include the development of electrodes, electrolyte and current collector layers and the development of printing inks based on environmentally benign carriers. Work is performed on the fundamental properties of materials used in the modules, including their mechanical properties at operating temperature, and the testing and post-test examination of modules, produced both in house and by partners.



*Planar fuel cell fabricated by silk screen printing. Fore-ground: translucent screen; background: printed cell layers. The magnification is approximately one to one.*

### Next generation of solid oxide fuel cells

The next generation of solid oxide fuel cells will have to obey the following criteria's:

- Low operation temperature (down to 550 °C)
- Redox stability, to allow for loss of fuel supply
- Direct combustion of methane
- Low cost
- Improved durability

A concentrated effort is needed to meet these criteria. Work has to be done to minimise losses that occur in different components in the fuel cell and to lower the operating temperature. The main benefits resulting from lowering of the operating temperature are lower cost and improved durability. Emphasis is to be put on the development and fabrication of improved oxygen electrodes, one of the main causes of internal resistance. It will also be necessary to use other types of electrolytes than the zirconia-based ones used in most solid oxide fuel cells today, for example ceria-based electrolytes. New types of anodes, which are stable under both reducing and oxidising conditions, are under development, based on materials that exhibit oxide, proton and electronic conductivity. For direct methane combustion, fuel electrodes need to be developed with a high catalytic activity for methane combustion, and a low tendency to form coke. The fuel electrodes for methane conversion will most likely be constructed from ceria-based materials coated with catalytic active species.

### The Membrane Project

Materials exhibiting both oxide ion and electronic conductivity may be used in dense form as oxygen separation membranes. As the gas transport takes place via ions the membranes are unlike porous membranes 100 % selective towards oxygen. Such dense membranes may find future use for oxygen production and production of synthesis gas. Synthesis gas (syngas) is a mixture of CO and H<sub>2</sub>. It is an intermediate product in industrially important processes like synthesis of ammonia and methanol. Today synthesis gas is produced via steam reforming or partial oxidation of methane. Using a mixed conducting membrane the oxidation process and the separation process (oxygen from air) may be integrated in one reactor. This route has the potential of becoming cheaper as well as more environmentally friendly than the existing routes.

The research activities in this field are carried out on commercial conditions for Haldor Topsøe A/S. Since 2000 the field of collaborators has further included Air Liquide (Fra), Snamprogetti (Ita) and Politecnico de Torino (Ita) within the frame-



*Jens Høgh mounting a ceramic membrane disk to be tested for its oxygen transport properties.*

work of an EU support project "Ceram gas". An important milestone for this project is a "proof of concept" experiment with a one meter long tube.

Among the candidate materials studied for syngas membranes are perovskites of the type  $\text{La}_{1-x}\text{Sr}_x\text{Fe}_{1-y}\text{Co}_y\text{O}_3$ . These materials are also studied for use in SOFC, and there is a great overlap also in characterisation techniques, material models and manufacturing processes between the SOFC projects and the membrane project resulting in a fruitful synergy between the projects.

The membrane research activities in the department covers investigation of fundamental properties like structure, oxygen non-stoichiometry and oxygen diffusion constant of candidate materials as well as more technological experiments like testing of membrane disks for syngas production. During 2001, a disk of one of the identified candidate materials was operated under syngas relevant conditions for 2 months revealing both high flux and excellent stability.

#### High temperature corrosion investigation

The lowering of the operational temperature in a SOFC stack has made it possible to use steel alloys as an interconnector material. Ferritic Fe-Cr alloys with ca. 22% Cr has proven particularly promising. For the past 2 years the oxidation of iron-chromium alloys has been investigated under conditions that resemble SOFC operational conditions. These studies have focused on:

- The influence of water as an oxidant
- Alloy composition
- Oxidation in reducing (anode) or oxidising (cathode) atmospheres

The oxidation resistance has been proven to be dependent on the alloy composition, where small additions of alloying elements have proven adequate. Furthermore, the oxidation resistance and/or scale morphology is greatly influenced by the oxidising atmosphere.

#### The fuel cell programme on line

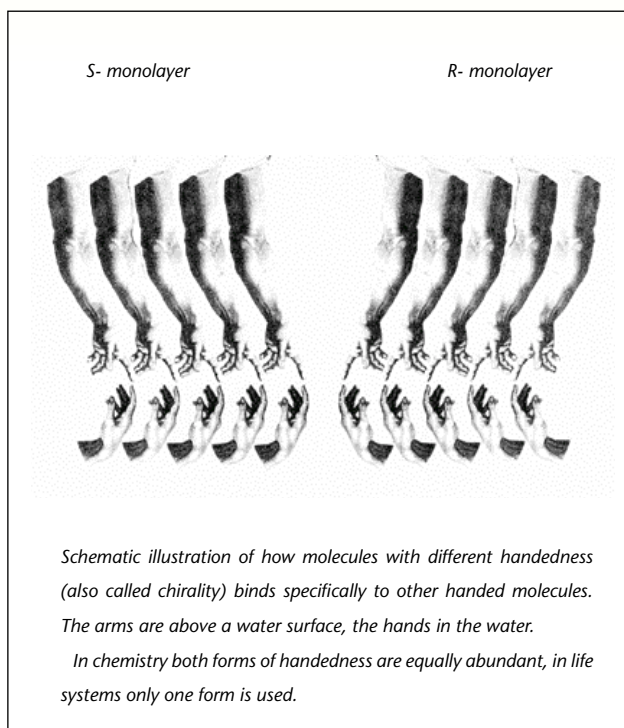
Further information about the Fuel Cell programme can be obtained from [www.SOFC.dk](http://www.SOFC.dk), as well as, through the Departments home page at Risø.

*S. Lindetoth*

## Biological Physics

The human genome is now solved. Next, remains the formidable task of understanding the meaning, the function, the structure and the place in the cells of the hundreds of thousand different proteins, which are coded for in the genome. The proteins are the machines of the cells. In the post genomic era a great deal of research and funding will be devoted to this kind of research to get a deeper understanding of life and how food and medicine works and can be improved. It is a new field of research, which demands collaboration between many traditionally separate disciplines, such as biology, chemistry and physics. The Department has been involved in several projects in this new frontier.

It is known that about 70% of all proteins resides in the membrane enclosing a cell. It contains proteins regulating transport of energy and matter in and out of the cell. It consists of two layers of lipids, which makes an internal oily layer about 4 nanometres thick, covered on both sides by very thin surfaces, to which water is attracted. Scattering techniques using reflections of neutrons or X-rays can give information about such membranes, which are supported by binding to a liquid or solid surface. In the simplest case, it is of interest to study just a single lipid layer, half a membrane, floating on a water surface.



Molecules and proteins soluted in water bind in specific ways to the formed interface. Most organic molecules are twisted (chiral) with a right- or left-handed turn. In chemical synthesis they are formed in equal amount. However, Pasteur found that the molecules of life were chiral in just one out of the two possible ways. It has been one of the major puzzles in biology to understand how such a selection could have occurred. The following extract from research results in the Department, shows that the properties at a water surface has the potential for selecting molecules of different chirality.

By adding a double layer to a half layer bound to a solid surface, all in water, a good model-cell-membrane can be formed. Neutron and X-ray reflection data gave accurate information about the layer thickness and distances for such a pure membrane. Similar measurements with an incorporation of Gramicidin showed the resulting changes in the cell wall thickness. That is an important antibiotic protein; when pairs are formed an ion pore is opened through the membrane.

In another line of research, theoretical models for proteins, their folding and function are studied in collaboration with a newly started basic research centre, "The Quantum Protein", at the Danish Technical University. Further theoretical collaboration is made with a group at the Niels Bohr Institute studying single proteins using optical tweezers. Collaborations with several other groups are also in progress. A special activity of the Department is the Graduate School for Biophysics, funded by Forskerakademiet. As a part of this activity an international summer school called "Physics of biomolecules and cells" was organized in Les Houches, France, 3-27 July. Some of the world's best scientists in the fields were lecturers. The course was three times overbooked and even students from elite-universities did apply in vain. Ten students from Denmark participated.

There is no doubt that the interest in biological systems and the physics thereof will continue to grow worldwide. At Risø some of the activities will be transferred to and concentrated in other departments in order to ensure a critical mass of expertise. The expertise in scattering technique and in theoretical models of fundamental phenomena remains strong in the Department.

*Per-Anker Lindgård*





*View towards Mont Blanc from the summer school in Les Houches.*

### Symmetry of molecules

Recent experiments bear on the question of pre-biotic mechanisms for selection of molecules of only one chirality. Monolayers of stearyl-lysine-thioethyl-ester, an amphiphilic derivative of the natural amino acid lysine, were prepared at the air-water interface using an equal mixture of left- and right-handed molecules. X-ray diffraction experiments in Hamburg at the Risø-built liquid-surface diffractometer showed that the 2D-crystallites formed at the interface contained the left- and right-handed monomers in equal amounts in a left/right ordered fashion. However, when the thus organized monomers were polymerised, the reaction proceeding along the 2D crystals (parallel to the water surface), short chains (oligomers) of a single handedness were formed in higher-than-stochastic abundance - by virtue of the 2D-lattice organization. Starting with the same monomers but using slightly unequal amounts of left- and right-handed

molecules, the left/right asymmetry was significantly enhanced among the longer oligopeptides formed. In experiments with different chiral monomers (stearyl-glutamic acid-N-carboxyanhydride), again starting with slightly unequal amounts of left- and right-handed molecules, the abundance of the predominant chirality was increased to nearly 100 pct. among the longer oligom the initial generation of a left-/right-handed imbalance of chiral amino acids at pre-biotic times generally lead to only very low imbalances. The mechanisms described here might be relevant for the conversion of such mixtures of amino acids to oligopeptides of a single handedness. Amphiphiles self-assembled at the air-water interface may have been highly relevant for pre-biology; also, the inherently lowered symmetry of the interface assists left/right symmetry breaking.

*Kristian Kjaer*



## Superconductivity

Superconductivity at high temperatures is a hectic field in science and technology. The driving force is the lacking understanding of the basic mechanism of high- $T_c$  superconductivity, and the prospects of a technological revolution if the full potential of the materials can be explored. Not least if the ultimate goal: a room temperature superconductor is developed. For many applications, especially within the energy sector, the key properties are the ability to conduct currents without loss and produce strong magnetic fields.

Basically superconductivity and magnetism are mutually exclusive phases, but they may co-exist. This interplay is generally observed in high- $T_c$  materials, which are antiferromagnetic insulators in the undoped form, and even at the optimal doping level for superconductivity (16%) antiferromagnetic fluctuations are found to survive. It is still subject to debate if these magnetic fluctuations are reminiscence of a competing antiferromagnetic ordering or they contribute to the formation of the superconducting state. Superconductors avoid the detrimental effect of magnetic fields by generating super-currents that exclude the magnetic flux from the interior or isolate it into vortices each containing one magnetic flux quantum. The vortices are strongly linked to the fundamental superconducting properties, and they are of significant importance for technological applications because they interact with transport currents and cause losses if not properly pinned. A case story elucidating the complex interplay between superconductivity, vortex structure and magnetism in  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$  is presented below.

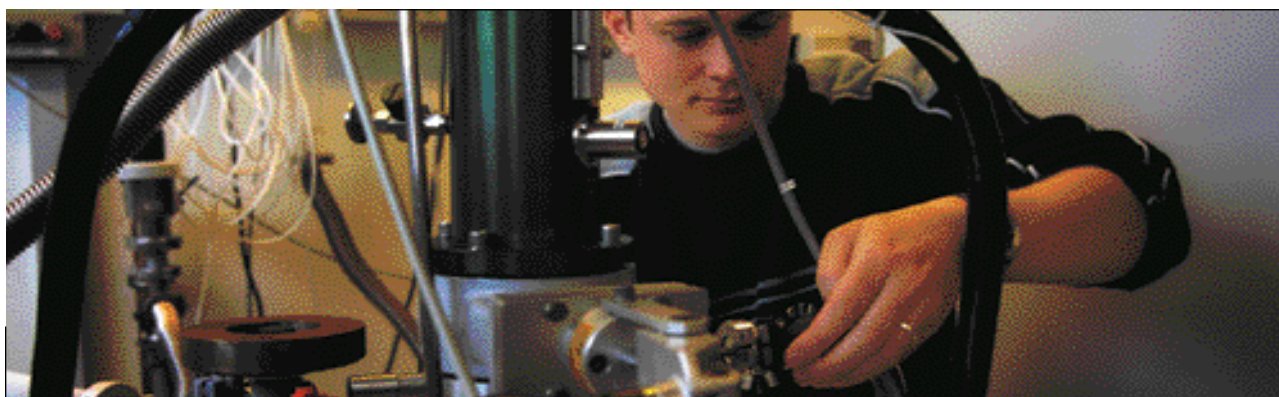
The general expertise in the Department on structural studies by neutron, synchrotron x-ray scattering and electron microscopy techniques is used in combination with bulk characterisation techniques to cover a broad range of activities on fundamental properties, materials synthesis and characterisation.

Superconductivity and magnetism co-exist in several of the  $\text{RENi}_2\text{B}_2\text{C}$  (RE = Rare Earth) materials. They are conventional superconductors but their basic properties are interesting for the general understanding of the competition between superconductivity and magnetism. For several years the magnetic phases and the vortex structure have been intensively studied in these materials. Evidence for the impact of magnetism on superconductivity has clearly been observed in  $\text{TmNi}_2\text{B}_2\text{C}$ , and recently it was shown that superconductivity perturbs the magnetic structure of  $\text{ErNi}_2\text{B}_2\text{C}$  at low temperatures where a small ferromagnetic moment develops.

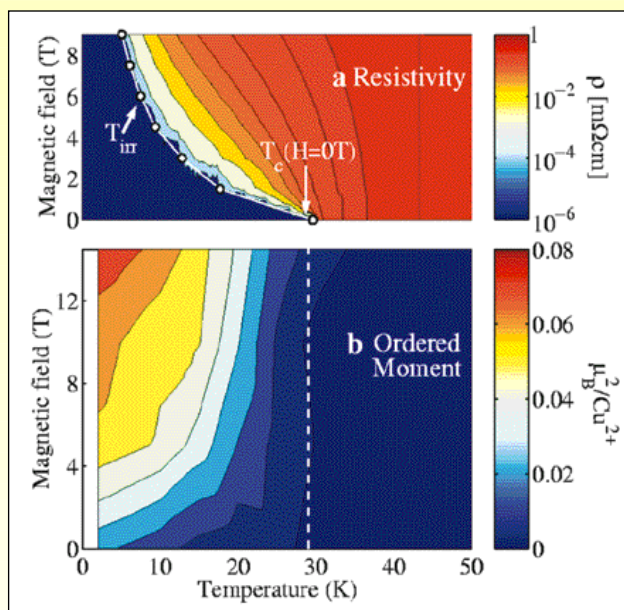
Recently initiatives were taken to start a synthesis programme on superconductors. Materials prepared include the newly discovered  $\text{MgB}_2$ , which is a traditional superconductor with an unexpected high critical temperature (39 K) and a significant potential for applications. A critical current of 52 A has been obtained in a  $\text{MgB}_2$ /steel tape with cross-section:  $4 \times 0.25 \text{ mm}^2$  at 4.2 K and 1 Tesla. Other interesting materials being synthesised are the so-called ladder compounds, that resemble the high- $T_c$  materials but the infinite copper-dioxide planes are split into ladders. Despite their lower dimensionality, they may be superconducting up to 80 K.

The Department has a close collaboration with the Danish company Nordic Superconductor Technologies A/S on development of the BiSCCO-2223/Ag high- $T_c$  superconducting tapes that have been used in the superconducting cable recently installed in the utility power net at the Amager Coupling station. In the past year focus has been on characterisation and synthesis of improved BiSCCO powders.

Niels Hessel Andersen



Master student Thomas Jørgensen eager to measure magneto-transport properties of high- $T_c$  superconductors.



Magneto-transport and neutron diffraction data for  $\text{La}_{1.9}\text{Sr}_{0.1}\text{CuO}_4$  as a function of temperature and magnetic field. (a) shows magneto-transport measurements parallel to the  $\text{CuO}_2$  planes; the colours indicate the level of the electrical resistivity. In a magnetic field, vortices are thought to form at temperatures where the resistivity falls below its value at  $T_c(H=0)$ . Phase coherent superconductivity, characterised by zero resistance, sets in at the much lower "irreversibility" temperature,  $T_{\text{irr}}(H)$ , marked by the white circles. The actual critical temperatures,  $T_c(H)$ , where the superconducting condensate disappears cannot be determined by the transport measurements. (b) shows the square of the ordered spin moment per  $\text{Cu}^{2+}$  ion as a function of temperature and applied magnetic field. The ordered moment squared is proportional to the observed neutron scattering signal. It first becomes significant below the zero-field superconducting transition temperature  $T_c(H=0)$ , and increases with decreasing temperature and increasing field. The data were taken at HMI, Berlin.

### Magnetism in the flux lines of $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$

One feature shared by all high-temperature superconductors is that they are of type-II. Thus, when they are placed in moderate magnetic fields the field lines penetrate the bulk to form a lattice of non-superconducting vortices around which supercurrents circulate. Understanding the structure and motion of vortices is a central problem in high- $T_c$  research, as it may provide important clues as to why these materials display superconductivity at such high temperatures.

Working with colleagues at NEC (USA), Oxford University (UK) and CEA (Grenoble, France), a group of researchers from the Department has recently made a number of key discoveries on the nature of the vortex state in the high-temperature superconductor  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$  (LSCO). Neutron scattering was used to study the interplay between magnetism and superconductivity in two members of the LSCO family. The first results for an optimally sample ( $x = 0.16$ ) showed that in the vortex state the magnetic field had the effect of strongly enhancing low-frequency magnetic fluctuations, which are otherwise destroyed by superconductivity (B. Lake et al. Science 291, 1759 (2001)).

This provided compelling evidence for one class of theoretical models, where superconductivity and magnetism are treated on an equal footing, which then compete at low-temperature. As the field is applied, vortices are created which, as they are non-superconducting, become small fluctuating nano-magnets.

From what was discovered in the first experiment, a second experiment was designed on a slightly under-doped crystal ( $x = 0.10$ ), where it was hoped to observe static magnetic order associated with the vortex state. The results of this experiment are summarised in the figure above. The top panel shows the electrical resistivity as a function of temperature and magnetic field, where it can be seen that in a field superconductivity is only achieved below the so-called irreversibility line where the vortices freeze to form a regular lattice. The bottom panel shows the neutron scattering results, where the main feature is a large increase in the magnetic signal as the field is increased. This again points to the inherently magnetic nature of the vortex state.

Des McMorrow

## Large Scale Facilities

The new Materials Research Department has considerable expertise in the use of X-ray and Neutron scattering requiring large-scale facilities. These techniques are used by a number of groups, and provide invaluable information on the nano-scale structure and dynamics of materials. Depending on the problem at hand, these techniques are capable of providing information in exquisite and unique detail. In 2001, x-ray and neutron scattering have been utilised to provide to questions of importance in science and technology, from the nature vortex matter in high-temperature superconductors to the kinetics of grain formation in metallic alloys.

One of the most significant events of 2001 in the Department's access to large-scale facilities was the signing of the agreement between Risø and the Paul Scherrer Institute (PSI) in Switzerland. PSI is situated just outside of Zurich, and operates SINQ, a powerful, accelerator based source of neutrons, which in the coming years is set to be one of the best sources in the world. Under the terms of the agreement, the Department will transfer three instruments to PSI in return for 50% of the available beamtime (some 20 weeks per instrument). The first instrument, RITA-II was transferred already in April 2001, and was commissioned in record time, allowing the first experiments to start in June. RITA-II is a new class of neutron spectrometer, which has been developed to provide information

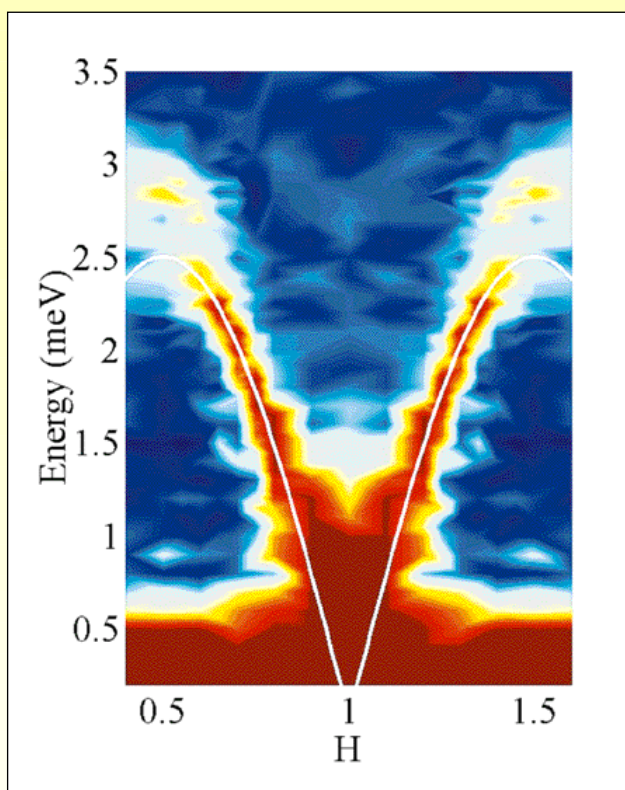
on the nano-scale dynamics of materials. One example of the type of investigation that has been undertaken with RITA-II is the study of the dynamics of magnetic nano-particles. Here the function of these materials in, for example, magnetic recording media, is determined by how these particles relax and exchange energy with their environment. Such relaxational processes can only be characterised fully and understood with neutron scattering. The second instrument, SANS (small angle scattering), will be shipped in April 2002, and will play a vital role, not only for the study of polymers, but also in many other fields, such as the structure of metallic alloys, high-temperature superconductors, etc.

When the third instrument, RITA-I, is shipped in 2003, Danish neutron scattering will have completed its move to its new base in Switzerland. The perspectives in this collaboration are great. Our Swiss partners are equally enthusiastic about the prospects for future collaboration. One measure of this was the first joint Swiss-Danish meeting on neutron scattering, which was held on November 16-17 and attracted over 60 participants. In addition to SINQ, the Swiss Light Source started operation in summer 2001, giving access to both neutrons and third-generation synchrotron radiation on one site.

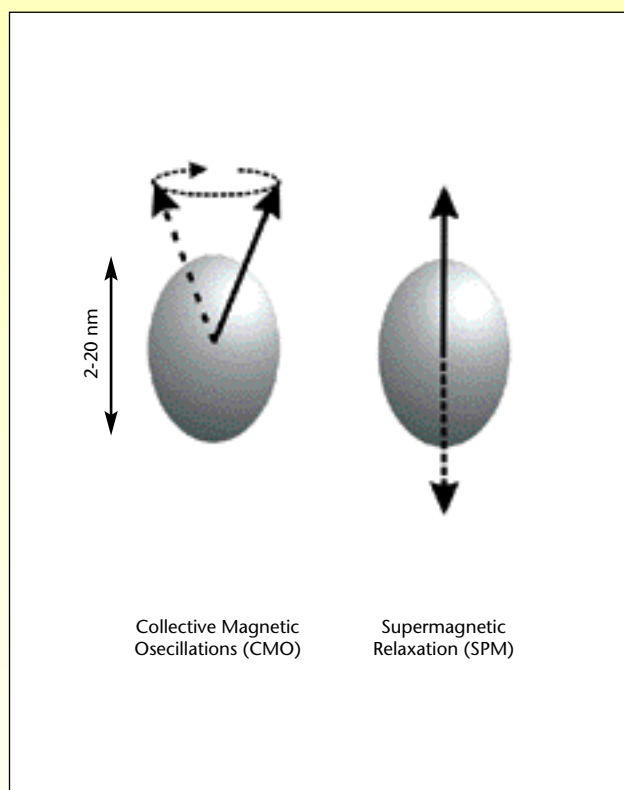
*Des McMorrow*



*Installation of RITA-II at PSI in May 2001. The spectrometer was moved to PSI in April, and was already taking its first data in June! Shown in the picture are Steen Bang and John Holm from AFM along with some colleagues from PSI. The instrument will be used to study fundamental magnetic properties of materials, including magnetic nanoparticles, superconductors, etc.*



The figure shows some surprising results from a recent experiment on a two-dimensional quantum magnet,  $K_2V_3O_8$  (Des McMorrow (Risø) and Stephen Nagler (Oak Ridge National, Laboratory)). This material is of interest because high-temperature superconductors are also two-dimensional quantum magnets, and the coupling of electrons which leads to superconductivity may in fact be produced by magnetic interactions. The solid line shows the expected energy dependence of the magnetic excitations, assuming that only nearest neighbours interact with each other. Clearly this model does not explain the extra intensity observed at higher energies, where the magnetic excitations actually split into two.



Relaxation modes of a ferromagnetic nanoparticle. The two different modes of magnetic dynamics in a ferromagnetic nanoparticle. In collective magnetic oscillations (CMO), the magnetisation precesses around the anisotropy axis of the particle, lowering the particle's magnetisation. In superparamagnetic relaxation (SPM), the magnetisation is spontaneously reversed by random thermal noise. Understanding and controlling the magnetisation dynamics is important for the application of nanoparticles in magnetic recording media. Inelastic neutron scattering is ideal for studying CMO and the high-temperature range of SPM. The RITA-II spectrometer is well suited to this task, as it can be set-up to have simultaneously both a high energy resolution and high neutron intensity.

### First experiments underway on RITA-II in Switzerland

Less than one year after the permanent closure of DR3, the first neutron scattering instrument to be moved to the Paul Scherrer Institute in Switzerland is producing science. RITA-II is a completely new type of neutron spectrometer, which allows the way in which electrons interact (or more strictly speaking, their spins) to be studied in unprecedented detail.

RITA-II will be used to study the properties of different types of materials, including high-temperature superconductors, mag-

netic nano-particles, etc. The fact that it was possible to install and commission successfully a brand new instrument in such a short period is tribute to a group of technicians in the Department, who could only complete the task by giving up lots of evenings and weekends. The second instrument, SANS, will be delivered to PSI at the start of next year, and the final one, RITA-I, in 2003.



## 22<sup>nd</sup> Risø International Symposium

The Symposium "Science of Metastable and Nanocrystalline Alloys - Structure, Properties and Modelling" which was held during five days in September had gathered about 80 participants from twenty countries. This is a field of growing activity world-wide and with a number of internationally leading scientists present at the Symposium, the participants contributed to a very successful meeting with many high quality talks and lively discussions. The contributions about metastable materials dealt in particular with alloys, which are amorphous and can be transformed into nanocrystalline structures. A number of talks concentrated on the special properties of nanocrystalline materials, for example their structure and

mechanical, thermal, chemical, electrical and magnetic properties. All the contributions from the Symposium were printed in a 500 page proceedings volume that is available from the Department. The proceedings can also be found on the web site [www.risoe.dk/afm/symp22/procelec.htm](http://www.risoe.dk/afm/symp22/procelec.htm).

The Symposium was organised by the Department in collaboration with the Engineering Science Centre for Structural Characterisation and Modelling of Materials at Risø.

*Morten Eldrup*



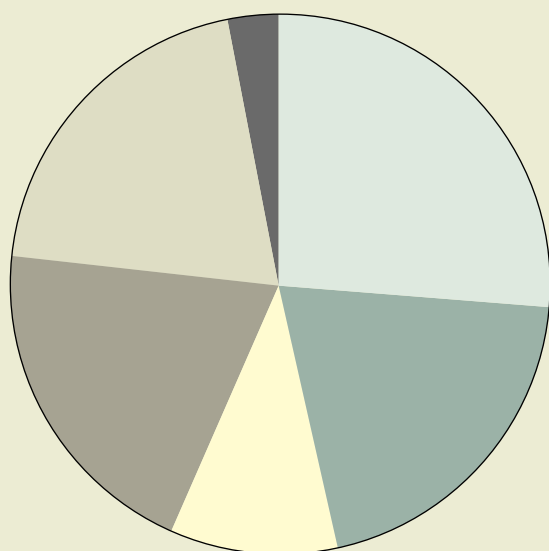
*Participants of the symposium looking for new directions of nano crystalline research during the excursion along the waterways in Copenhagen. All possibilities are explored.*

# Finances

The activities of the Department are supported by a combination of government funding, focused project funds from national, international or EU research programmes, and fully

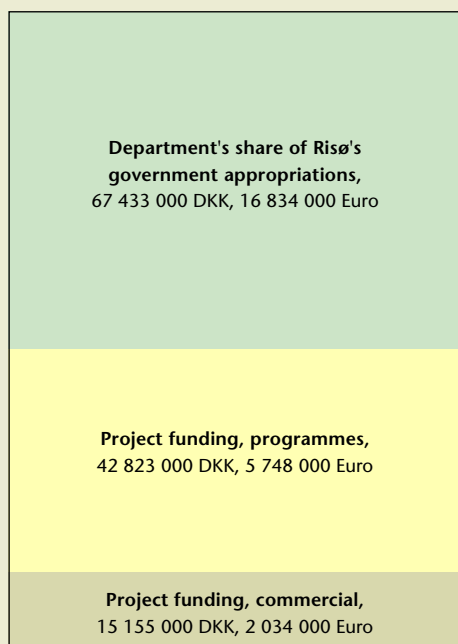
commercial contracts. The numbers are given in Danish kroner and the equivalent amounts in Euros are also shown.

**Project Income**



- Commercial contracts,**  
15 155 000 DKK, 26%
- Other programmes,**  
11 879 000 DKK, 20%
- Danish Energy Research Programme,**  
5 883 000 DKK, 10%
- EU,**  
11 572 000 DKK, 20%
- Danish Research Councils,**  
11 742 000 DKK, 20%
- Danish National Research Foundation,**  
1 746 000 DKK, 3%

**Income**



**Total:** 125 411 000 DKK, 16 834 000 Euro

**Expenditures**



**Total:** 124 198 000 DKK, 16 671 000 Euro

# Staff

A person leaving (#) or joining (\*) the Department during 2001 is counted as 1/2 man year.

## Head of Department

Feidenhans'l, Robert

## Programme Heads

Andersen, Niels Hessel

Brøndsted, Povl

Juul Jensen, Dorte

Leffers, Torben

Lilholt, Hans

Linderoth, Søren

Pedersen, Allan Schrøder (Deputy Head)

Singh, Bachu

## Academic staff (47 man years)

Andersen, Svend Ib #

Bang-Møller, Søren \*, #

Bentzen, Janet J.

Bilde-Sørensen, Jørgen B.

Bonanos, Nicholas

Borring, Jan

Borum, Kaj K.

Carstensen, Jesper Vejtlø

Clausen, Kurt Nørgaard

Debel, Christian P.

Eldrup, Morten

Flyvbjerg, Henrik K.

Friehling, Peter B.\*

Gamstedt, Kristofer #

Gerstenberg, Michael

Grievet, Jean-Claude

Hansen, Kent Kammer \*

Hendriksen, Peter Vang

Huang, Xiaoxu

Jacobsen, Torben K. #

Johansen, Bjørn S.

Jørgensen, Mette Juhl #

Kjær, Kristian

Larsen, Peter Halvor

Lassen, Niels C. Krieger #

Lebech, Bente

Lefmann, Kim

Lindgård, Per-Anker

Liu, Yi-Lin

Lybye, Dorte

Lystrup, Aage S.

Løgstrup Andersen, Tom

Margulies, Lawrence \*

McMorrow, Desmond F.

Megnis, Modris \*

Menon, Moha

Mikkelsen, Lars Pilgaard \*

Mogensen, Mogens

Nielsen, Karsten Agersted \*

Nielsen, Mourits #

Nilsson, Tage M.

Pantleon, Wolfgang

Pedersen, Ole Bøcker

Poulsen, Finn Willy

Poulsen, Henning F.

Poulsen, Jørgen

Primdahl, Søren #

Pryds, Nini H.

Sørensen, Bent F.

Thomsen, Heidi Sjølin \*

Toft, Palle

Toftegaard, Helmuth L.

Wang, Wei Guo \*

Wert, John A.

Winther, Grethe

## Postgraduate students (19 man years)

Abrahamsen, Asger B.

Andersen, Lotte Gottschalck #

Andreasen, Jens W.

Christiansen, Jesper #

Christensen, Niels Bech

Dinesen, Anders Reves

Glerup, Marianne #

Hansson, Anette Nørgaard \*

Højerslev, Christian

Jensen, Karin Vels

Klausen, Stine Nyborg

Koch, Søren

Larsen, Axel W.\*

Madsen, Bo

Lauridsen, Erik Mejdal

Mikkelsen, Lars

Nørbygaard, Thomas #

Toft, Katrine Nørgaard

Pedersen, Trine Bjerre

Petersen, Kenneth #

Poulsen, Mette \*

Schmidt, Henrik Nikolaj Blicher  
 Vegge, Tejs #  
 Thygesen, Anders \*

#### Post Docs (9.5 man years)

Bowen, Jacob R.\*  
 Bunk, Oliver \*  
 Cendre, Emanuelle  
 Hasimoto, Shin-Ichi \*  
 Hatchwell, Charles E.  
 Knudsen, Jesper \*  
 Margulies, Lawrence #  
 Nielsen, Søren Fæster  
 Ramousse, Severine  
 Rozlosnik, Noemi \*  
 Sabin, Tanya #  
 Schmidt, Søren  
 Zhang, Erlin  
 Åstrand, Per-Olof

#### Technical staff (39 man years)

Abdellahi, Ebtisam  
 Adrian, Frank  
 Bang, Steen  
 Breiting, Bjarne O.  
 Christiansen, Gitte \*  
 Frederiksen, Henning K.  
 Gravesen, Niels N.  
 Hansen, Anna K.\*  
 Hansen, Erik G. \*  
 Holm, John H.  
 Iversen, Jette \*  
 Jensen, Knud  
 Jensen, Palle V. +  
 Jespersen, John  
 Kjøller, John  
 Klitholm, Cliver  
 Larsen, Bent #  
 Larsen, Bent L.  
 Larsen, Jan #  
 Larsen, Kjeld J. C.  
 Lindbo, Jørgen #  
 Mikkelsen, Claus  
 Nielsen, Palle H.  
 Nielsen, Steen Erik  
 Nilsson, Helmer  
 Olesen, Preben  
 Olsen, Benny F.  
 Olsen, Henning

Olsen, Ole E.  
 Olsson, Jens O.  
 Paulsen, Henrik  
 Pedersen, Hanne \*  
 Pedersen, Niels Jørgen  
 Rasmussen, Ove  
 Sandsted, Kjeld  
 Saxild, Finn B.  
 Skaarup, Per  
 Stenkilde, Pia S.  
 Strauss, Torben R.  
 Sørensen, Carsten G.  
 Sørensen, Erling  
 Udbjerg, Charlotte L.  
 Vogeley, Erik  
 Aagesen, Svend #

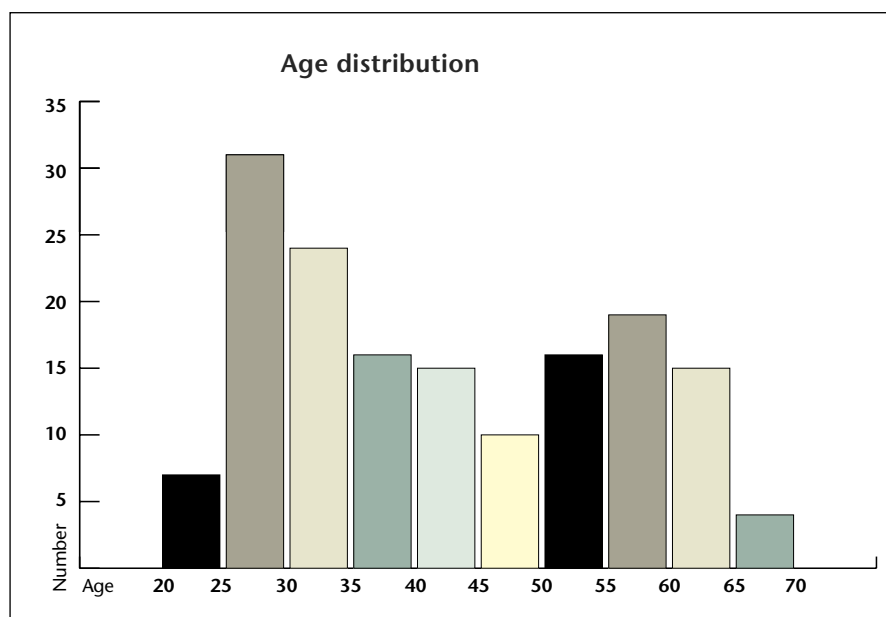
#### Office staff (5.5 man years)

Larsen, Ann \*  
 Lauritsen, Grethe Wengel  
 Nielsen, Jytte Pihl  
 Sørensen, Eva M.  
 Thomsen, Ann  
 Voss, Anita

#### Apprentices (1.5 man years)

Kheder, Shwan\*  
 Madsen, Christian H.

+) deceased August 2001





## Educational work

The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate courses and research projects, in collaboration with universities and industry, to one-day courses for high school classes. In addition, many staff members of the Department act as external university examiners. The education of Ph.D. students is one of the most important educational activities, and during 2001 6 Ph.D. students finished their projects, while 18 Ph.D. projects were still ongoing.



*Crystallography is not always easy. Ph.D. student Asger Abrahamsen and M.Sc. student Susan Blak Nyrup are trying to find the atomic planes in a crystal lattice.*

### Ph.D. projects finished during 2001

#### **Lotte Gottschalck Andersen**

"Structural properties of superconducting BiSCCO/Ag tapes during annealing". Technical University of Denmark. Supervisors: Claus Schelde Jacobsen (DTU), Henning Friis Poulsen

#### **Marianne Glerup**

"Raman spectroscopy and X-ray diffraction on oxides at high temperature". University of Copenhagen. Supervisors: Rolf W. Bert (DTU), Ole Faurskov Nielsen (KU), Finn Willy Poulsen

#### **Erik Mejdal Lauridsen**

"Nucleation and grain growth studies by use of a 3D X-ray microscope". University of Copenhagen, Denmark. Supervisors: Jens Als-Nielsen (KU), Henning Friis Poulsen, Dorte Juul Jensen

#### **Thomas Nørbygaard**

"Structure and characteristics of general and special grain boundaries". University of Copenhagen, Denmark. Supervisors: Erik Johnson (KU), Jørgen B. Bilde-Sørensen

#### **Kenneth Petersen (industrial project)**

"Development of the spray-forming process for production of steel-based composites". Technical University of Denmark. Supervisors: Jan Lauberg List (Danish Steel Works) Stuart Clyens (DTI), Knud Aage Thorsen (DTU), Allan Schrøder Pedersen

#### **Tejs Vegge**

"Defect dynamics at the atomic scale". Technical University of Denmark. Supervisors: Karsten Wedel Jacobsen (DTU), Torben Leffers, Ole Bøcker Pedersen

## Ongoing Ph.D. projects

### **Asger Abrahamsen**

"Magnetic properties of superconductors". Technical University of Denmark. Supervisors: Docent, lic.scient. Claus Schelte Jacobsen (DTU), Niels Hessel Andersen

### **Jens W. Andreasen**

"Nano-structured catalysts". Technical university of Denmark. Supervisors: Kenny Ståhl (DTU), Frank Berg Rasmussen (HT A/S), Robert Feidenhans'l

### **Jesper Christiansen**

"Dislocation interactions with surfaces and grain boundaries". Technical University of Denmark. Supervisors: Jakob Schiøtz, Karsten Wedel Jacobsen (DTU), Torben Leffers

### **Niels Bech Christensen**

"Quantum phase transistions". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Des Mc Morrow

### **Anders Reves Dinesen**

"Magnetic properties of amorphous and nanocrystalline alloys". Technical University of Denmark. Supervisors: Steen Mørup (DTU), Søren Linderøth, Nini H. Pryds

### **Anette Nørgaard Hansson**

"Protection against metallic oxidation". Technical University of Denmark. Supervisors: Marcel Somers (DTU), Søren Linderøth, Mogens Mogensen, Peter Friehling

### **Christian Højerslev**

"The influence of microstructure on the fatigue properties of high strength materials for cold forging tools". Technical University of Denmark. Supervisors: Marcel Somers (DTU), Povl Brøndsted, Jesper Vejlø Carstensen

### **Karin Vels Jensen**

"Interface in composite electrodes for SOFC and high temperature electrolyzers". Technical University of Denmark. Supervisors: Ib Chorkendorff (DTU), Mogens Mogensen, Jørgen B. Bilde-Sørensen

### **Stine Nyborg Klausen**

"Magnetic dynamics of nanoparticles". Technical University of Denmark. Supervisors: Steen Mørup (DTU), Kim Lefmann, Kurt N. Clausen

### **Søren Koch**

"Contacting of ceramic materials". Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Peter Vang Hendriksen, Mogens Mogensen

### **Axel W. Larsen**

"Nucleation in metals". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Henning Friis Poulsen, Dorte Juul Jensen

### **Bo Madsen**

"Evaluation of properties of aligned plant fibre composites – an experimental modelling study". Supervisors: Lars Damkilde (DTU), Preben Hoffmeyer (DTU), Anne Belinda Thomsen (PBK, Risø), Hans Lilholt

### **Lars Mikkelsen**

"Oxidation of iron-chromium alloys". University of Southern Denmark, Odense. Supervisors: Eivind Skou (USD), Søren Linderøth, Mogens Mogensen, Peter Halvor Larsen

### **Katrine Nørgaard Toft**

"Magnetic properties of superconductors". University of Copenhagen, Denmark. Supervisors: J. Jensen (KU), Niels Hessel Andersen

### **Trine Bjerre Pedersen**

"Modelling of residual stresses in sprayformed structures". Technical University of Denmark. Supervisors: Jesper Hattel (DTU), Nini Pryds

### **Mette Poulsen**

"Nanostructuring with bonding". Technical University of Denmark. Supervisors: Flemming Jensen (DTU), Robert Feidenhans'l

### **Henrik Nikolaj Blicher Schmidt**

"Modelling of mechanical and metallurgical properties of Friction Stir Welded joints". Technical University of Denmark. Supervisors: Jesper Hattel (DTU), John A. Wert

### **Anders Thygesen**

"High performance hemp fibres and improved fibre network for composites (HeFiNaC)". Royal Veterinary and Agricultural University, Copenhagen, Denmark. Supervisors: Per Ole Olesen (KVL), Claus Felby (KVL), Hans Lilholt

## Published work

The number of publications in 2001 was 223. Some papers are co-authored with scientists from other institutions, as a result of national and international collaboration. Names of authors from the Department are written in italics.

International Publications	31
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Danish Publications	39
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Publications in Proceedings	40
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Popularised Scientific Publications	43
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## International Publications

1. Alonso, C.; Eliash, R.; Jensen, T.R.; Kjær, K.; Lahav, M.; Leiserowitz, L.; Guest intercalation at corrugated surface of host monolayer crystal on water: Cholesteryl-L-glutamate and water-soluble amino acids. *J. Am. Chem. Soc.* (2001) v. 123 p. 10105-10106
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26. Eldrup, M.; Singh, B.N.; Void nucleation in fcc and bcc metals: A comparison of neutron irradiated copper and iron. *Mater. Sci. Forum* (2001) v. 363-365 p. 79-81
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33. Gamstedt, E.K.; Östlund, S.; Fatigue propagation of fibre-bridged cracks in unidirectional polymer-matrix composites. *Appl. Compos. Mater.* (2001) v. 8 p. 385-410
34. Gao, D.C.; Yang, W.Y.; Huang, X.; Sun, Z.Q.; Superplastic deformation in a coarse-grained  $\text{Fe}_3\text{Al}$  based alloy. *Scripta Mater.* (2001) v. 44 p. 501-505
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38. Glerup, M.; Sørensen, G.O.; Beichert, P.; Johnson, M.S.; Bonding in *cis*- and *trans*-dichlorobis(dimethylsulfide)platinum(II) studied by vibrational (micro)spectroscopy and force field calculations. *Spectrochim. Acta A* (2002) v. 58 p. 129-139
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To promote an innovative and environmentally sustainable technological development within the areas of energy, industrial technology and bioproduction through research, innovation and advisory services.

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Risø's research shall extend the boundaries for the understanding of nature's processes and interactions right down to the molecular nanoscale. The results obtained shall set new trends for the development of sustainable technologies within the fields of energy, industrial technology and biotechnology. The efforts made shall benefit Danish society and lead to the development of new multi-billion industries.

Risø's activities in 2001 are reported in the following publications: Risø Annual Report (available in Danish and English), Risø's Annual Performance Report (Danish) and the annual progress reports of the seven research departments (English). All publications and further information can be obtained from [risoe.dk](http://risoe.dk). Printed publications are available from the Information Service Department, tel.: +45 4677 4004, e-mail: [risoe@risoe.dk](mailto:risoe@risoe.dk), fax: +45 4677 4013.



